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SIZE EXCLUSION CHROMATOGRAPHY FOR CHARACTERIZATION OF HEAVY OIL/BITUMEN VACUUM BOTTOMS AS FEEDSTOCKS FOR COPROCESSING

P.M. Rahimi and J.F. Kelly

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by

P.M. Rahimi* and J.F. Kelly**

ABSTRACT

Cold Lake vacuum bottoms, a potential feedstock for coprocessing, has been separated according to molecular weight on Styragel 100 Å using preparative gel permeation chromatography (GPC). The average molecular weight (determined by vapour pressure osomometer) of 10 arbitrary fractions obtained by this fractionation technique ranged from 228 to 2074. Using an analytical GPC equipped with Shodex columns (divinyl benzene polymer) a linear relationship was found between the log $\bar{M}n$ of fractions and retention volumes. Elemental analysis of the fractions showed an increase in nitrogen and sulphur contents as a function of molecular weight. H/C atomic ratios increased with decreased molecular weight and peaked at 648 g/mol. Conversely, aromaticity decreased sharply to a minimum at 548 g/mol and then increased gradually as molecular weight increased.

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UTILISATION DE LA CHROMATOGRAPHIE PAR EXCLUSION GRANULOMÉTRIQUE POUR CARACTÉRISER DES DISTILLATS DE PÉTROLE LOURD ET DE BITUME COMME CHARGE D'ALIMENTATION POUR LE COTRAITEMENT

par

P.M. Rahimi* et J.F. Kelly**

RÉSUMÉ

Des distillats de Cold Lake, représentant une charge d'alimentation potentielle pour le cotraitement, ont été séparés d'après leur masse moléculaire sur un Styragel 100 A au moyen de la chromatographie de perméation sur gel (CPG). La masse moléculaire moyenne (déterminée au moyen d'un osmomètre à pression) de dix fractions arbitraires obtenues par cette technique de fractionnement, variait de 228 à 2 074. En utilisant un CPG (chromatographe de perméation sur gel) de précision doté de colonnes Shodex (polymère de vinyl-benzène), on a découvert un rapport linéaire entre le log $\bar{M}n$ des fractions et celui des volumes de rétention. Une analyse élémentaire des fractions à démontré que leur teneur en azote et en soufre augmentait en fonction de leur masse moléculaire. Les rapports atomiques H/C ont augmenté en fonction de la diminution de la masse moléculaire pour atteindre un sommet à 648 g/mol. Inversement, l'aromaticité a diminué rapidement jusqu'à un minimum de 548 g/mol pour ensuite augmenter graduellement à mesure que la masse moléculaire prenait de l'importance.

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INTRODUCTION

It can be expected that coprocessing heavy oil/bitumen and coal will be one of the viable processes for future synthetic fuel production in Canada. Results obtained from coprocessing Cold Lake vacuum bottoms and Forestburg subbituminous C coal indicated high distillate yields and coal conversion (1-4). A comparison of these results with those from liquefaction of the same coal in hydrogenated anthracene oil showed distinct advantages for coprocessing (4). Recently, researchers at HRI using petroleum residues as coprocessing media concluded that coprocessing has economic advantages over coal liquefaction (5).

In light of recent new developments in liquefaction, it seems appropriate to develop an easy and reliable routine characterization technique for coprocessing solvents. Hydrocarbon type analysis is a widely used technique in the petroleum industry for chemical characterization of feedstocks and products (6-8). Most laboratories use high performance liquid chromatography (HPLC) for class separation. A reliable method for hydrocarbon type separation was developed in the USBM-API Project 60 (9) and was modified at CANMET (10).

In recent years there has been increasing effort to use gel permeation chromatography (GPC) to characterize coal-derived liquids and feedstocks (11-19). GPC has also been used to characterize Athabasca bitumen (20), to determine the molecular weight of Athabasca bitumen (21) and to characterize and determine the molecular weight of Athabasca aspnaltenes (22). Although GPC is a very useful technique for analysis of complex mixtures, accurate quantitative determination presents a number of problems. Problems associated with GPC in analyzing complex mixtures of coal-derived liquids or bitumens are summarized below.

1. GPC separates molecules according to their size in solution. To relate molecular size (weight) to retention volume (time) requires calibration of the GPC column. What compounds are to be used for calibration? It has been shown that each homologous series of compounds has its own size-molecular weight relationship (21,23). This problem may partially be solved for coal-derived liquids by using narrow molecular weight cuts of coal-derived liquids as standards. However, Kahn (16) has shown that

this method is very specific and calibration should be done for each coal-derived liquid obtained under different conditions.

2. Molecules having completely different molecular weight elute at the same retention volume. This problem may be solved by using a highly polar solvent such as pyridine.
3. Detectors used with most HPLC systems are one or more of the following: UV, visible, and IR photometers, differential refractometers (RI), conductance bridges, transport detectors and fluorimeters. Solvents used with these detectors should be chosen so no interference occurs between the sample and the solvent. Moreover, each detector should be calibrated with each of the compounds to be determined because each compound has a different sensitivity which depends on its chemical structure. The response may not always vary linearly with changes in concentration. This means that each compound should be calibrated separately. For coal-derived or coprocessing liquids, where a large number of compounds are present in the mixture, the separation and isolation of individual compounds is impossible. Ford and Kennard (24) showed that the evaporative analyzer (EA) detector (25) is not sensitive to the chemical structure of low molecular weight polymers which produced almost equivalent responses when passed through the instrument. These results indicated that the EA detector is a pure mass detector and can be used for quantitative determination of compounds that are difficult to isolate for calibration purposes.

In spite of the above difficulties and problems, GPC can be extremely as a first step useful for characterizing complex mixtures. In this study, preparative GPC was used to separate Cold Lake vacuum bottoms, a potential solvent for coprocessing, into different fractions which were further characterized using analytical GPC, elemental analysis and ^{1}Hnmr.

EXPERIMENTAL

Samples of Cold Lake vacuum bottoms were obtained from the Strathcona refinery of Imperial Oil Ltd. The gross characteristics of this solvent are shown in Table 1.

Preparative GPC was carried out on a Waters LC 500 equipped with refractive index and a 122 x 2.5-cm column packed with styragel 100 A. The bitumen was injected in a mixture with THF (0.153 g/cc). THF was used for GPC elution at a rate of 12.5 cc/min and the fractions were collected at intervals as shown in Fig. 1. The fractionation (3 mL of sample) was repeated several times to obtain enough material for analysis. Analytical GPC separation was performed on a Waters M 6000A solvent delivery system equipped with refractive index and dual channel UV detectors and three Shodex columns (divinyl benzene polymer) 1X A-803 and 2X A-802 in series.

The average molecular weight of separated fractions was determined using a Knauer osmometer at 40°C in benzene.

Chromatographic solvent (THF) was passed through a 0.2-μm fluoropore filter before use. THF was obtained from Caledon Laboratories Ltd. Benzene was used as received from Anachemia Ltd.

Carbon, hydrogen and nitrogen analyses were performed on a CHN 240 Perkin Elmer analyzer and sulphur analyses were performed on a Leco analyzer.

^{1}Hnmr analyses were performed on a Varian CFT-20 Fourier transform spectrometer. $CDCl_3$ was used as a solvent and internal lock.

RESULTS AND DISCUSSION

GPC SEPARATION OF CLVB

The weight percentage yields of the fractions separated by preparative GPC (Fig. 1) are shown in Table 2. Fractions 1 to 5 constitute approximately 71 wt % of the total sample so that the gross properties of the total CLVB depends on their chemical compositions. The analytical GPC of total CLVB is shown in Fig. 2. Both UV and RI responses consist of two peaks - high and low molecular weight components. The RI and UV responses of the 10 fractions are shown in Figs. 3 and 4 respectively. It appears that the elution of each fraction follows the molecular weight, i.e., the higher the molecular weight the shorter the retention time. Moreover, there is a linear relationship between retention volume and the logarithm of the number average molecular weight (Fig. 5). It has been shown (21) that for total Athabasca bitumen using styragel columns, the relationship between retention time and molecular weight is not linear below 500 g/mol. Using Shodex columns, the linearity

seems to nold even below 300 g/mol. No attempt was made to measure the molecular weight of the fractions by analytical GPC since a previous study on Athabasca bitumen fractions (21) has shown that the response of each fraction to UV and RI detectors does not depend only on concentration but also on its chemical structure.

Molecular Weight Distribution of CLVB Fractions

The molecular weight distribution of CLVB fractions separated by preparative GPC and measured by VPO in benzene is shown in Table 2. The weight per cent of each fraction was measured after evaporating solvent (THF) under vacuum. Altnough this method of collecting samples and determining molecular weight is very time consuming, it is more reliable than analytical GPC because of tne problems associated with the GPC method as discussed earlier. Figure 6 shows what can be considered tne correct molecular weight distribution of CLVB.

Elemental Analysis of CLVB Fractions

Tne elemental composition of tne 10 CLVB fractions collected using preparative GPC is shown in Table 2. Since fractions 1 to 5 constitute the bulk of material, it is expected that the chemical properties of the total CLVB would depend mainly on these fractions. The nitrogen content of these fractions increased linearly with molecular weight (Fig. 7). However, the sulphur content dropped slightly then increased as molecular weight increased (Fig. 8).

The per cent of saturated hydrogen (aliphatic, naphthenic and benzilic) as determined by ^{1}Hnmr is shown in Table 2. Poor peak separation prevented accurate quantitative determination of each type of hydrogen however, the percentage of total saturated hydrogen decreased as molecular weight decreased. H/C atomic ratio as a function of molecular weight is shown in Fig. 9. The H/C ratio of fractions increased sharply with increasing molecular weight and peaked at $\bar{M}n$ 648, then decreased gradually to 1.36 at $\bar{M}n$ 2074. As shown in Table 2, a high percentage of CLVB fractions have relatively high H/C ratios. It has been previously reported tnat CLVB is a good hydrogen donor (26). Presumably, fractions 2 to 6 are better hydrogen donors than tne others.

Aromaticities of CLVB Fractions

Aromaticities of CLVB fractions were determined using the Brown-Ladner treatment and are shown in Table 2. As shown in Fig. 10 aromaticity of these fractions decreased sharply to a minimum as molecular weight increased and then increased gradually. As might be expected, the minimum in aromaticity (Fig. 10) coincides with the maximum in H/C ratio (Fig. 9). The decrease in aromaticity as a function of molecular weight indicates an increase in side chains of hydrocarbons up to $\bar{M}n$ 648 and then aromatization (higher fa) takes place.

Comparison of Athabasca Bitumen with CLVB

Comparison of the structural parameters of the fractions of CLVB with those of Athabasca bitumen obtained from ^{1}Hnmr and ^{13}Cnmr indicated that the two bitumens are similarities (27). In Table 3, a comparison of the chemical composition of CLVB and Athabasca bitumen (20) is based on the molecular weight of different fractions. Cold Lake fractions having molecular weights similar to those of Athabasca bitumen also have a similar elemental composition. H/C atomic ratios of Athabasca bitumen fractions are slightly higher than for Cold Lake. Cold Lake fractions have higher sulphur at higher molecular weights, and it appears that this trend is reversed at lower molecular weights. The nitrogen content of both bitumen fractions at higher molecular weights is very similar whereas at lower molecular weights Athabasca bitumen fractions have a higher nitrogen content.

SUMMARY

The objective of this work was to demonstrate the feasibility of using preparative GPC for characterization of heavy oil/bitumen vacuum bottoms as potential feedstocks for coprocessing. The results are summarized below:

1. Preparative GPC is an effective easy technique for separating CLVB into its components according to their molecular weights.
2. Using analytical GPC and Shodex columns, CLVB fractions were eluted according to their molecular weights, i.e., the higher the molecular weight the shorter the retention time.

3. It may be possible to use analytical GPC in conjunction with a mass analyzer detector, which is not sensitive to the structural changes in the molecules, to determine molecular weight.
4. In this work true molecular weight distribution of CLVB was obtained by collecting fractions and weighing.
5. It appears that a linear relationship exists between the molecular weight of fractions and nitrogen content.
6. Sulphur is concentrated in heavy fractions.
7. H/C atomic ratios of the fractions increased with decreased molecular weight and then decreased as molecular weight increased.
8. Aromaticity of the fractions dropped sharply as molecular weight increased from 228 to 648. Further increase in molecular weight resulted in a slight increase in aromaticity.
9. Similar molecular weight fractions of Cold Lake vacuum bottoms and Athabasca bitumen have similar H/C ratios.
10. The differences in the elemental composition of Cold Lake vacuum bottoms and Athabasca bitumen fractions, which have similar molecular weights, are relatively small.

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Table 1 - Characteristics of Cold Lake vacuum bottoms

General	
Specific gravity 15/15°C	1.038
Conradson carbon residue, wt %	17.1
Pentane insolubles (asphaltenes), wt %	23.48
Benzene insolubles (preasphaltenes), wt %	0.2
Aromaticity (^{1}Hnmr)	34.5
Viscosity at 80°C, poise	249.12
100°C, poise	39.40
110°C, poise	21.59
Molecular weight (VPO), g/mol	820.0
Distillation (Spinning Band method)	
IBP, °C	420
Distillate (-525°C), wt %	16.75
Residue (+525°C), wt %	83.25
Elemental analysis (wt %)	
Carbon	78.6
Hydrogen	9.3
Sulphur	5.5
Nitrogen	0.6
Ash	0.0
Oxygen (by difference)	5.9
Metal content, ppm	
Ni	93
V	235
Fe	18

Table 2 - Elemental analysis, molecular weight and aromaticities of CLVB fractions

Fraction No.	Yield wt %	$\bar{M}n$[1] g/mol	C	H	N	S	% H[2] sat.	H/C[3]	fa[4]
			wt %						
1	12.5	2074	80.2	9.1	0.78	6.7	93.8	1.36	36.2
2	20.7	1753	79.5	9.5	0.68	6.0	95.0	1.44	31.6
3	15.3	988	80.7	10.2	0.48	4.9	94.7	1.52	28.0
4	12.0	648	79.4	10.3	0.38	4.3	95.0	1.55	26.3
5	10.3	522	78.5	9.9	0.32	4.3	93.2	1.52	29.1
6	8.2	439	77.4	9.4	0.31	4.4	91.0	1.46	33.5
7	6.9	366	76.4	8.8	0.22	4.4	90.3	1.38	37.7
8	4.7	370	78.0	8.4	0.39	4.8	88.5	1.30	42.5
9	3.4	329	76.8	7.8	0.29	4.5	83.2	1.23	48.8
10	5.8	228	69.7	6.6	0.21	4.0	81.3	1.14	53.6

(1) Molecular weights were determined using VPO.

(2) Per cent saturate hydrogen to total hydrogen, determined by ^{1}Hnmr.

(3) H/C atomic ratio.

(4) Determined by Brown-Ladner treatment.

Table 3 - Comparison of chemical composition of CLVB and Athabasca bitumens at similar molecular weights

			C	H	N	S		No. of S and N atoms per one molecule	
		$\bar{M}n$	wt %				H/C	S	N
A)	CLVB	1753	79.5	9.5	0.68	6.0	1.44	3.3	0.85
	Athabasca	1680	77.1	9.4	0.70	5.3	1.50	2.8	0.84
B)	CLVB	648	79.4	10.3	0.38	4.3	1.55	0.87	0.17
	Athabasca	710	81.7	11.0	0.30	3.7	1.60	0.80	0.16
C)	CLVB	366	76.4	8.8	0.22	4.4	1.38	0.50	0.05
	Athabasca	370	79.0	8.9	0.40	6.3	1.40	0.70	0.11

CAPTIONS

Fig. 1 - Preparative GPC separation of Cold Lake vacuum bottoms

Fig. 2 - Analytical GPC separation of Cold Lake vacuum bottoms

Fig. 3 - GPC sequence of elution of Cold Lake vacuum bottoms fractions, RI response

Fig. 4 - GPC sequence of elution of Cold Lake vacuum bottoms fractions, UV response

Fig. 5 - Shodex column calibration curve for Cold Lake vacuum bottoms fractions

Fig. 6 - Molecular weight distribution of Cold Lake vacuum bottoms

Fig. 7 - Nitrogen content of Cold Lake vacuum bottoms fractions versus molecular weight

Fig. 8 - Sulphur content of Cold Lake vacuum bottoms fractions versus molecular weight

Fig. 9 - H/C atomic ratios of Cold Lake vacuum bottoms fractions versus molecular weight

Fig. 10 - Aromaticity versus molecular weight distribution of Cold Lake vacuum bottoms fractions

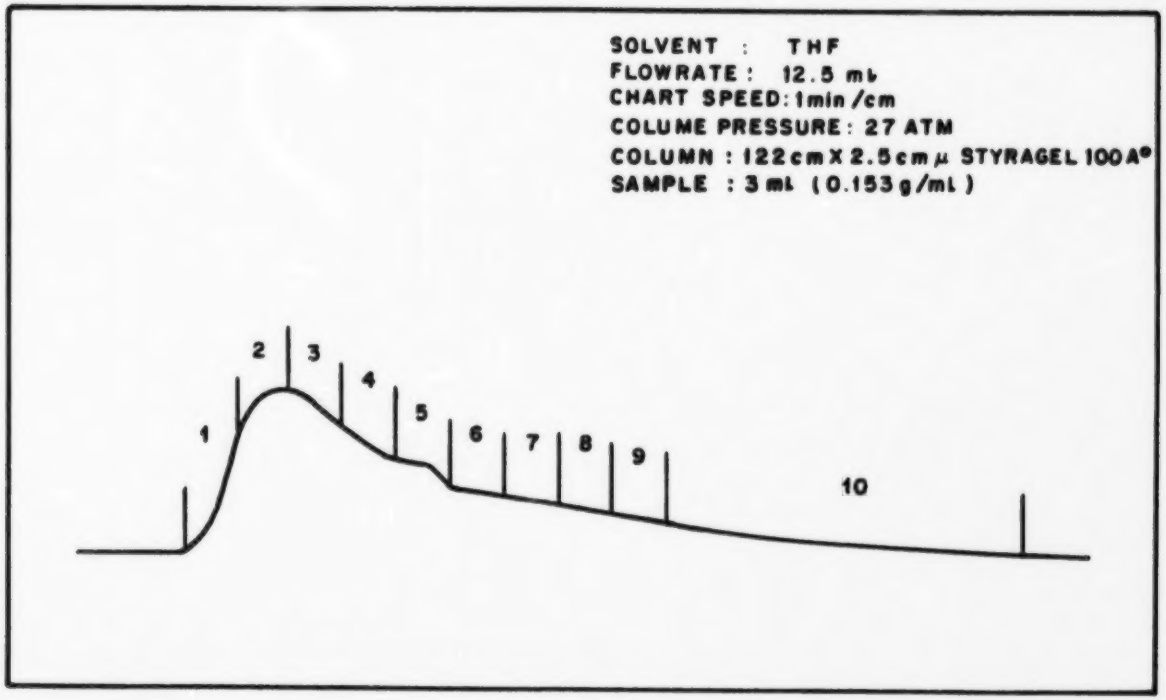


Figure 1 - Preparative GPC separation of Cold Lake vacuum bottoms

SAMPLE : 5μl
COLUMN : SHODEX A-803S, 2X A-802S IN SERIES
SOLVENT : THF
FLOW RATE : 2 ml/min
COLUME PRESSURE: 1000 psi
DETECTOR: UV 254 nm, 2.0 AUFS; RI 8X
CHART SPEED: 1.5 min/cm

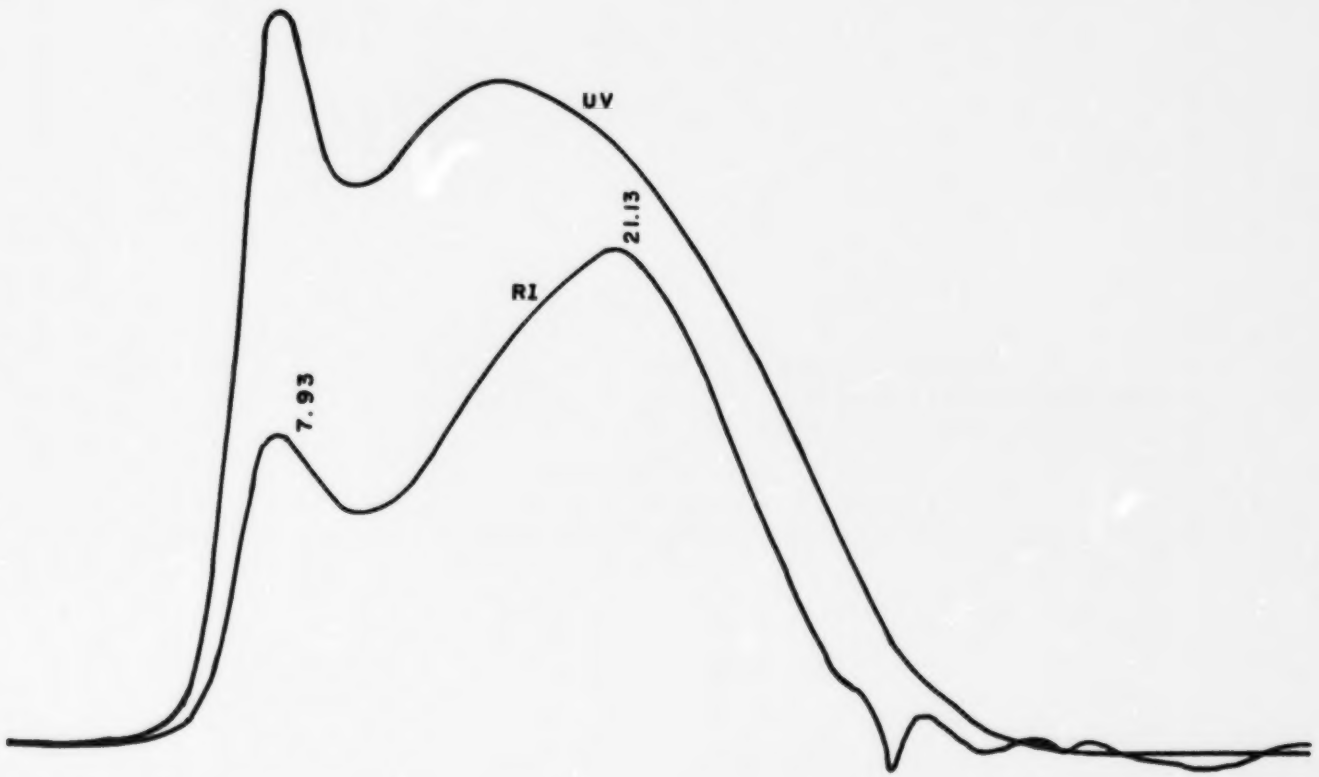


Figure 2 - Analytical GPC separation of Cold Lake vacuum bottoms

Figure 3 - GPC sequence of elution of Cold Lake vacuum bottoms fractions. RI response

SAMPLE : 10 µl
COLUMN : SHODEX A-803S, 2X A 802S IN SERIES
SOLVENT : THF
FLOW RATE : 2 ml/min
COLUMN PRESSURE : 1000 psi
DETECTOR : UV : 254 nm, 2.0 AUFS
RI : 8X
CHART SPEED : 1.5 min/cm

PEAK	RETENTION TIME	
1	8.00 ,	9.73
2	8.06 ,	10.40
3	8.26 ,	11.60
4	8.33 ,	11.66
5		11.73
6		12.26
7		12.46
8		12.53
9	12.53	13.60
10	12.53	13.60

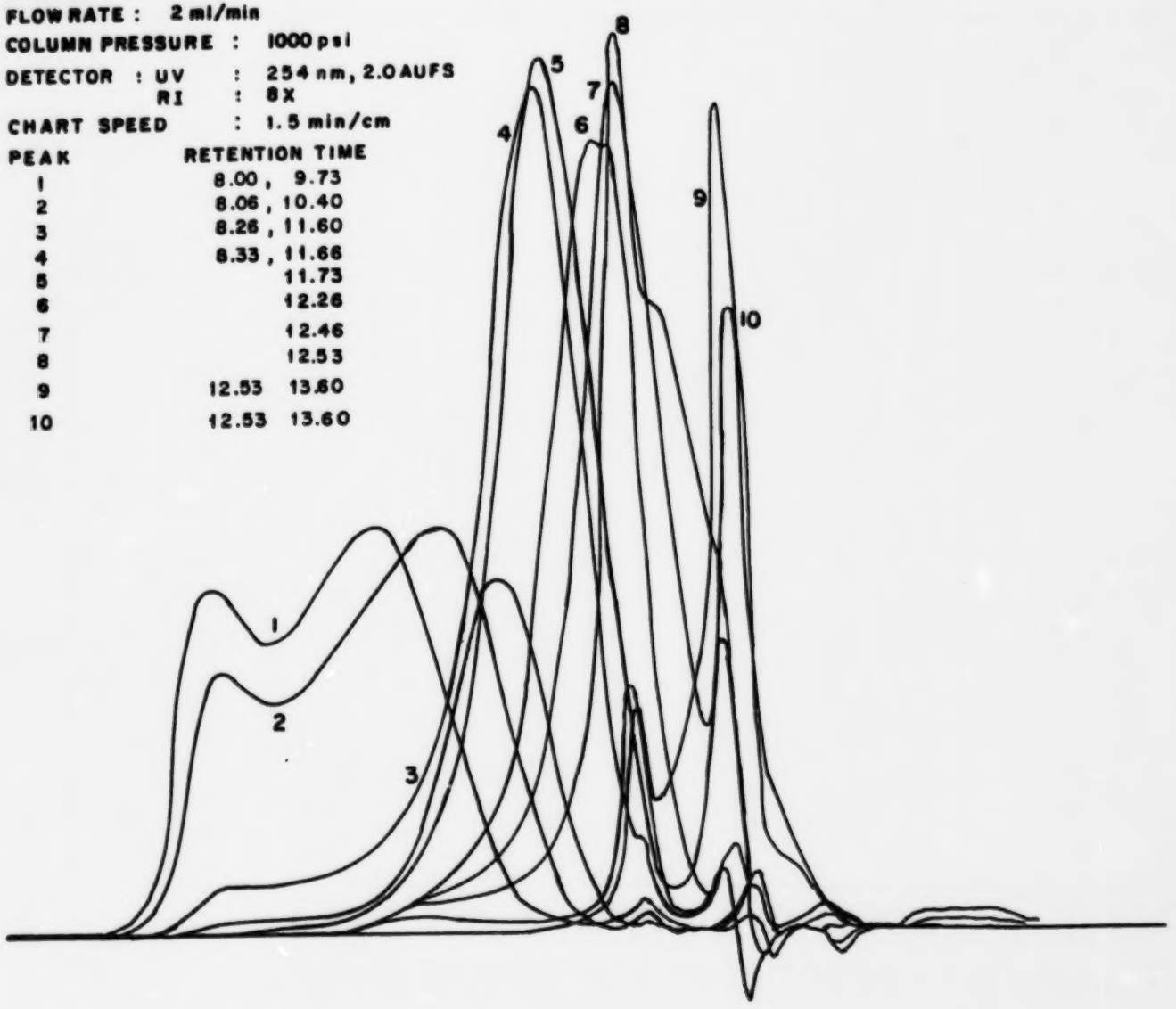
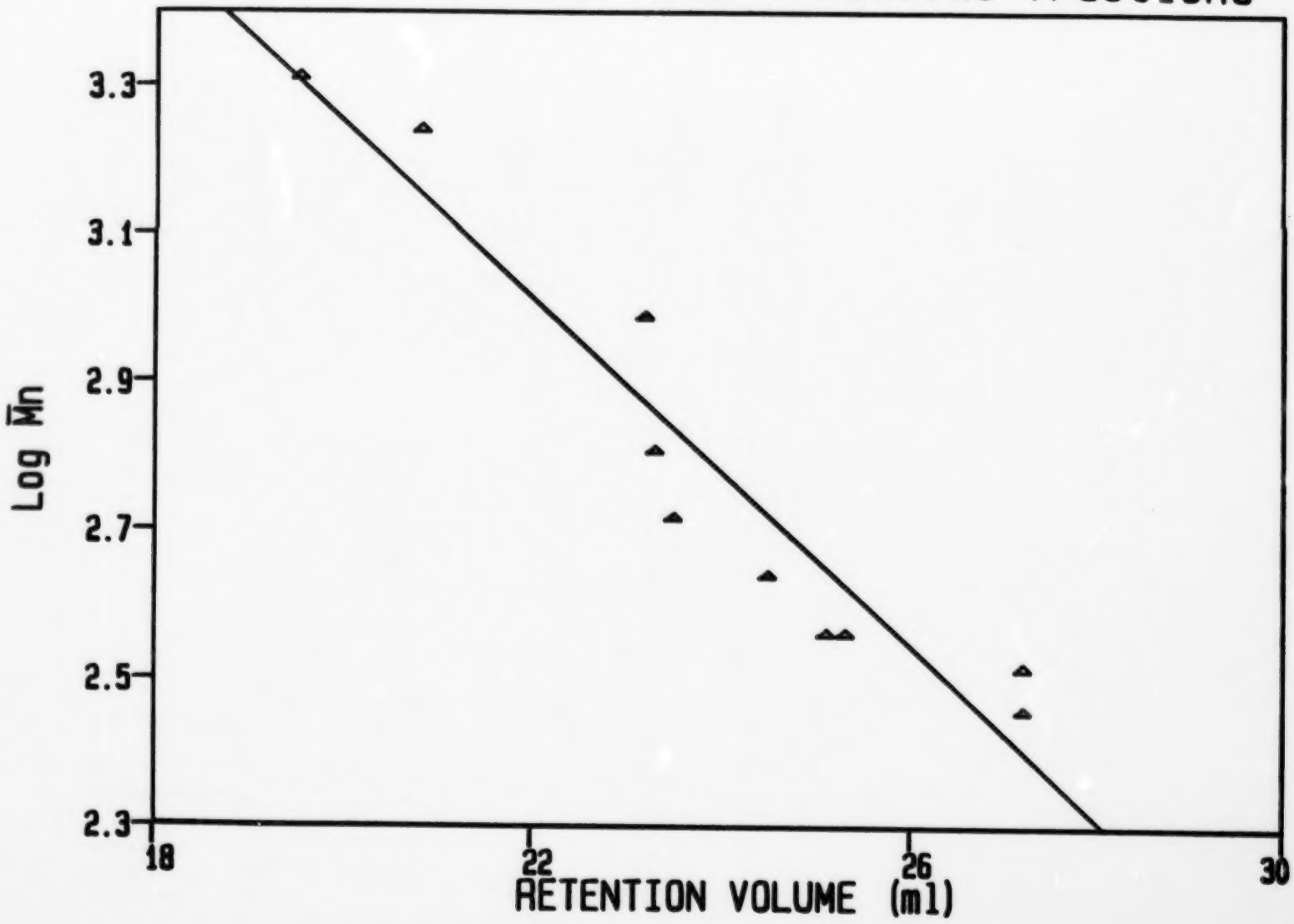


Fig.5. Shodex column calibration curve for Cold Lake vacuum bottoms fractions



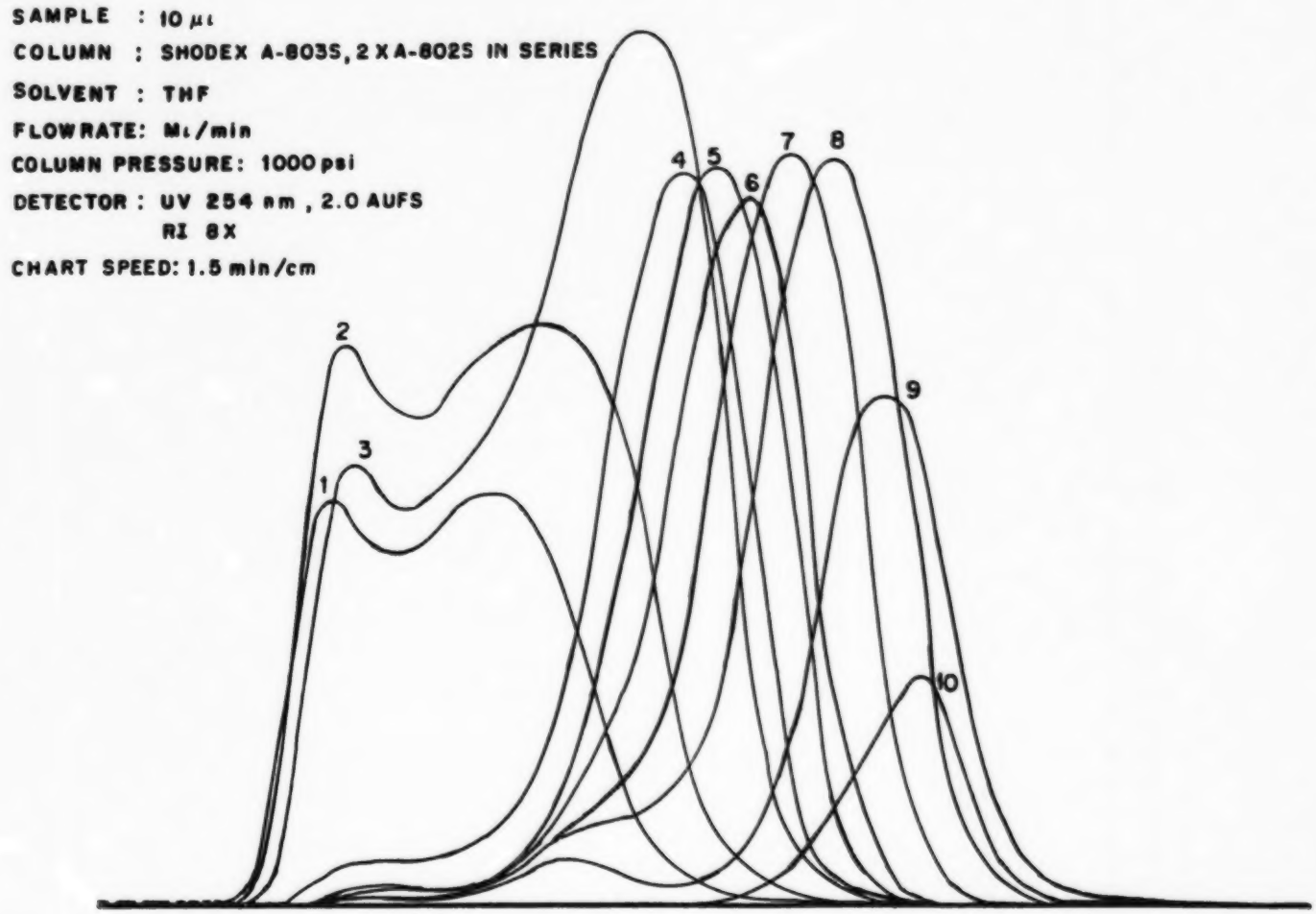


Figure 4 - GPC sequence of elution of Cold Lake vacuum bottoms fractions. UV response

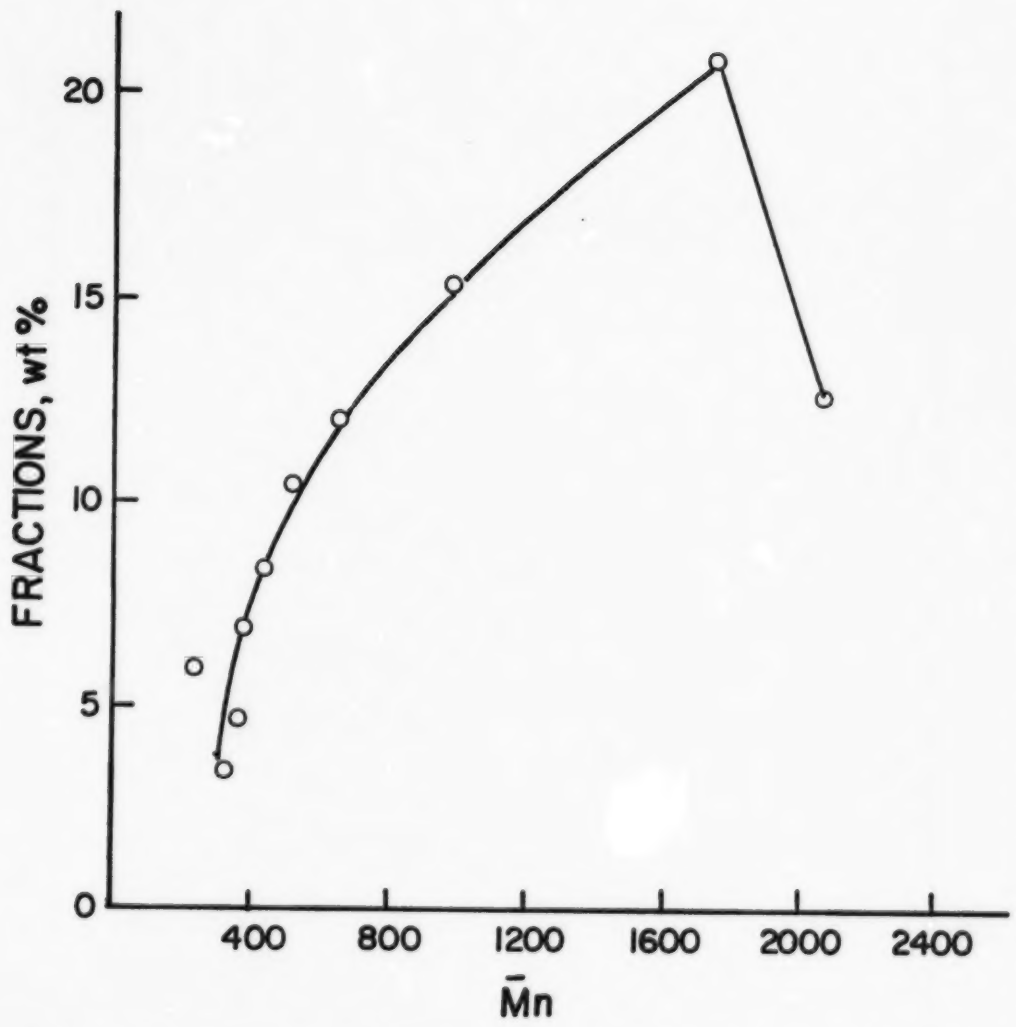


Figure 8 - Molecular weight distribution of Cold Lake vacuum bottoms

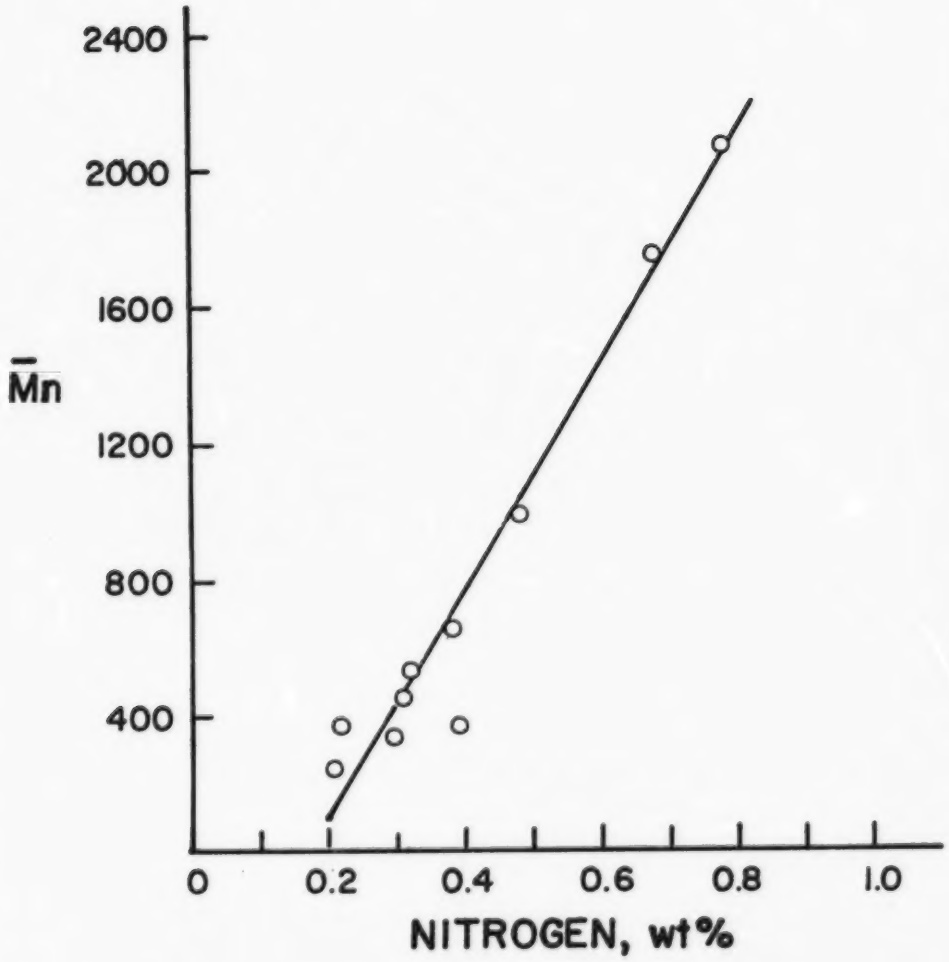


Figure 7 - Nitrogen content of Cold Lake Vacuum bottoms fractions versus molecular weight

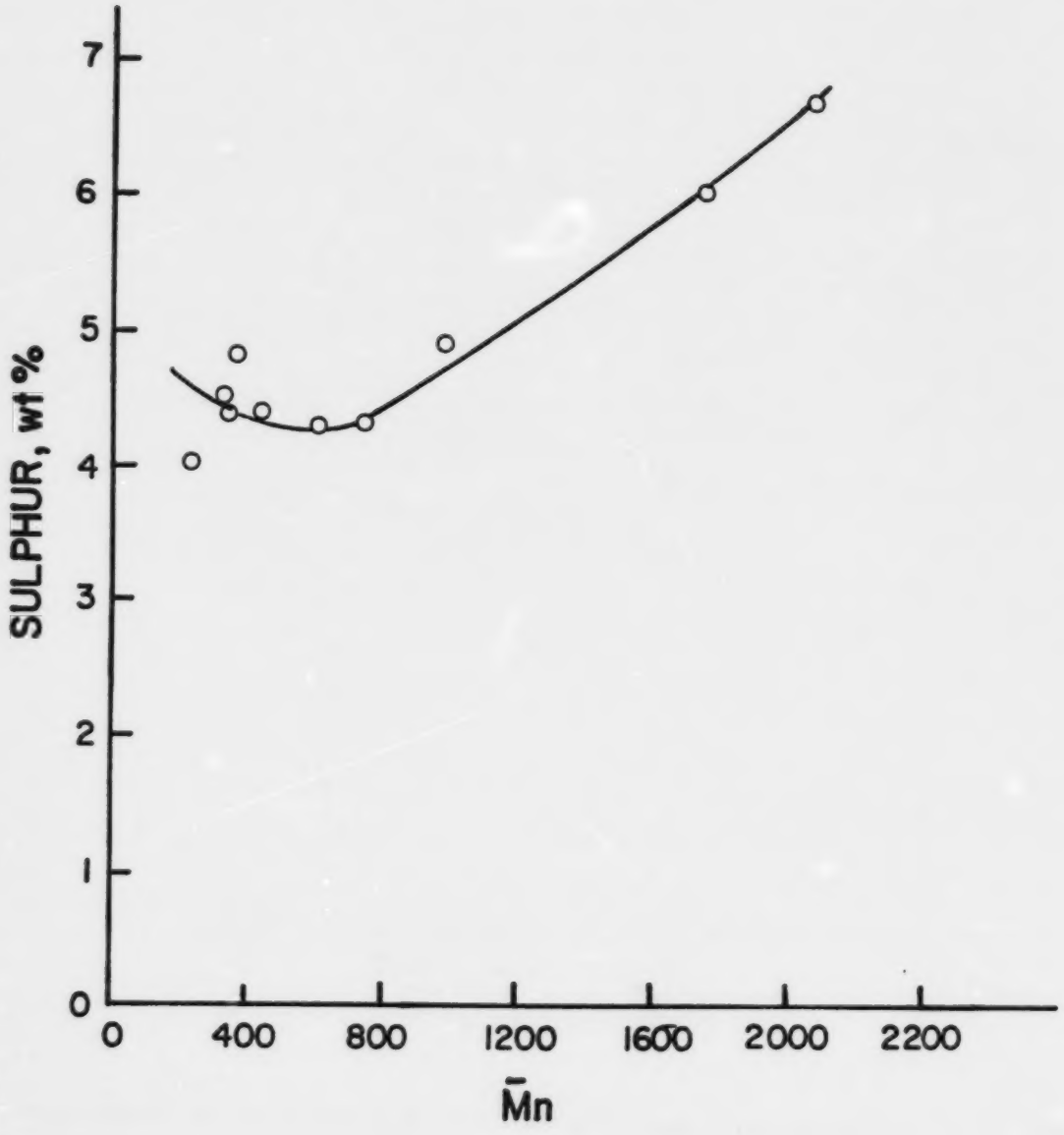


Figure 8 - Sulphur content of Cold Lake Vacuum bottoms fractions versus molecular weight

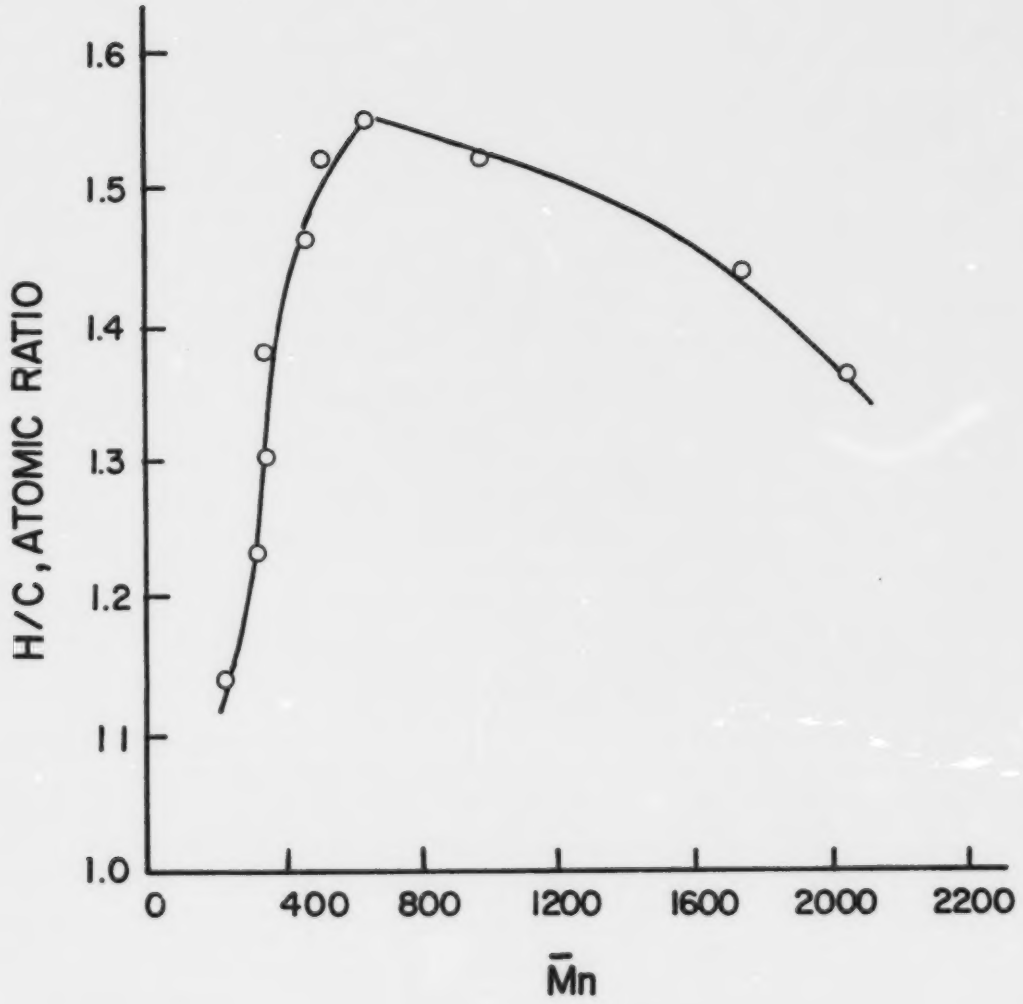


Figure 8 - H/C atomic ratios of Cold Lake vacuum bottoms fractions versus molecular weight

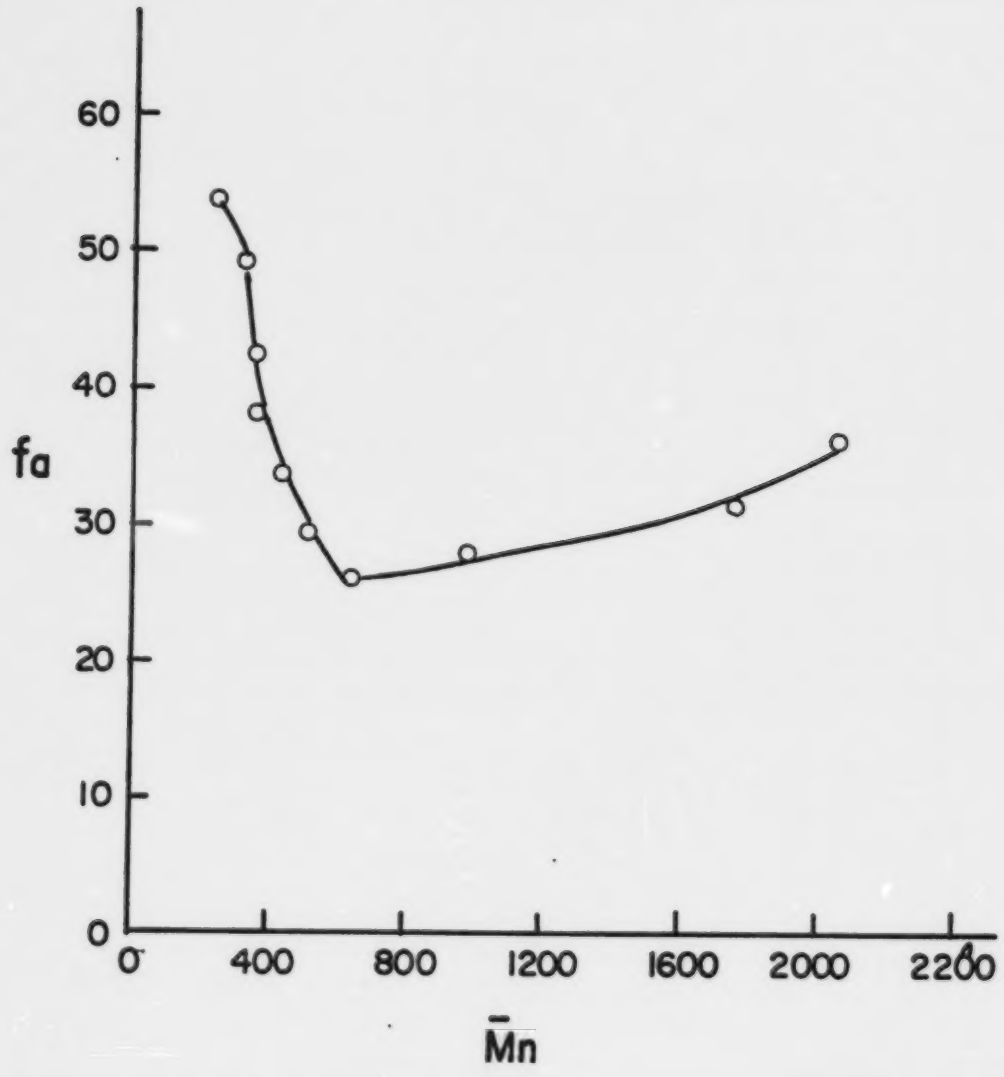


Figure 10 - Aromaticity versus molecular weight distribution of Cold Lake vacuum bottoms fractions